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FRG: BMFT's 1988 Interim Report on Materials Research Program

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ADVANCED MATERIALS

Description of Program Areas—Ceramics

[Text]

This section deals with monolithic ceramics and includes ceramic coatings. It is primarily concerned with high performance structural ceramics, but also encompasses some functional ceramics.

Ceramics have been used as an industrial material for decades (e.g., ceramic insulators). The new and increased industrial demands of the 1970's gave prominence to ceramics as modern high performance materials. Attention was focused on their potential for technological and industrial applications, based on both the physical properties (electrical resistance, ferroelectric properties, ionic conductivity etc.) of ceramics, and their thermal resistance, corrosion and abrasion resistance, biocompatibility, and relatively low specific gravity. The high temperature superconductivity of ceramics was added to this list in 1986.

Figure 1: Ceramics Compared with Superalloys, Source: Hoechst Ceramtech [not reproduced]

Figure 2: Ceramics Compared with Other Materials. (Specific strength (strength to weight ratio) is shown as a function of temperature). Source: MTU [not reproduced]

The development of ceramic components for use at high operating temperatures and under high dynamic stress was the goal of both the subprogram on "Ceramic Components for Automobile Gas Turbines" that formed part of the BMFT [Federal Ministry of Research and Technology] program on "Raw Materials Research 1976-1984" and the program area concerned with the manufacture and testing of ceramic components for gasoline and diesel engines. The ceramics funding in the Materials Research Program, with its focus on ceramic structural materials, was established in 1985 as the continuation and extension of earlier funding areas. The main result of these preliminary programs was success in proving the feasibility in principle of both an automobile gas turbine with its most important high-temperature components in ceramics, and ceramic components in gasoline and diesel engines. The development of these components led to discoveries that have opened applications for modern ceramic engineering materials with great market potential in the fields of energy technology, process engineering, metallurgy, and engine construction. Some examples are the components shown in Table 1, although these are primarily used at low dynamic stress.

Table 1: Ceramic Components From Earlier BMFT Programs

Component	Material	Use
Bearing rings	Silicon carbide (SiC)	Pumps, bearings
Burner components	Silicon carbide(SiC)	Welding, domestic and industrial burners
Port liners	Silicon nitride (Si_3N_4)	
Piston heads	Aluminum titanate (Al_2TiO_5)	Combustion engines
Heat exchangers	Zirconium oxide (ZrO_2)	
Vertical pipes, conveyer pipes, mixers	Silicon nitride (Si_3N_4)	Power engineering
	Silicon carbide (SiC)	
Jet nozzles	Aluminum titanate (Al_2TiO_5)	Metallurgy
	Silicon carbide (SiC)	
	Silicon carbide (SiC)	Sandblasting, etc.

The Materials Research Program thus had significant results and know-how on which to build.

Basic Materials and Process Development

[Text] Research on structural ceramics and ceramic reinforcement is particularly important. High performance ceramics are usually multiphase systems. Knowledge of the number, chemical composition, and limits (temperature, pressure) of the various material phases is important both in the production of ceramic materials and for their properties. The kinetics of phase formation is also important. This area of structural research is of fundamental significance for the material production process and for both the properties and the application limits of the materials. Computer-aided studies on the

structure of ceramic materials began in 1986 and the initial results of these studies were patentable. For example, zones of stability were calculated and experimentally confirmed in the silicon nitride (Si_3N_4), silicon carbide (SiC), and nitrogen (N_2) system as a function of temperature and nitrogen pressure. This had direct technical consequences, for example on pressure and temperature control in hot isostatic pressing (HIP) of SiC whisker-reinforced Si_3N_4 .

The FRG occupies a leading international position in structural research. However further comprehensive studies are necessary.

The second area of interest is structural reinforcement, an important subject in ceramic materials and process development. The two major objectives pursued here are

improving sturdiness and fracture resistance. Much work has been done on these topics within the Materials Research Program since 1985. Included are reinforcement processes such as alloying (Si alloys, SiC alloys, etc.), crystallization of glass phases, phase transition reinforcement, particle-, fiber-, and whisker-reinforcement, and multiple reinforcement. Table 1 provides a simplified overview of failure mechanisms and possible countermeasures in the case of high performance ceramic materials. Most of these measures involve structural reinforcements. The effect of titanium carbide (TiC) deposition in silicon carbide ceramics exemplifies the results of efforts in this area. Initial results show that the resilience of approximately 400 MPa could be increased to 720 MPa. The fracture resistance was increased from $3 \text{ MPa m}^{1/2}$ to $6.1 \text{ MPa m}^{1/2}$ as a result of crack deflection in the crack-particle interaction.

Understanding the various failure mechanisms and their interaction under complex stress is one of the most difficult of the basic problems facing ceramics research and it is the key to countermeasures. Work on structural reinforcement in the Materials Research Program should therefore be intensified in the future.

Another area of interest is materials characterization and testing. An example of development here is the thermoshock device, which allows control of the temperature cycle under tension and is equipped with an acoustic emission device to study the crack formation and propagation phase. Apart from testing resistance to thermal shock, this development permits a rapid, economic characterization of the sturdiness of ceramic materials at high temperatures.

Despite many advances, the problem of analysis and testing procedures has by no means been adequately solved. At present, nondestructive ceramics testing cannot recognize fracture-inducing faults with sufficient certainty. The further development of destructive testing methods and fracture mechanics is also needed to obtain sufficiently precise material characteristics for ceramics under the desired conditions of use so that safer components may be designed.

Priority Program of the German Research Association (DFG)

[Text] The DFG has been provided with funds for this program by the BMFT. Thirty-eight projects will begin in 1988.

The "High Performance Ceramic Materials" program is intended to encourage not only chemical engineers,

physicists, ceramicists, mineralogists, and materials scientists, but also experts in processing methods and materials testers to cooperate on joint projects. Their differing approaches, methods, and experiences can, in most cases, contribute much more significantly to problem-solving on a joint, interdisciplinary basis than on an individual basis.

The program is therefore also an important step forward in the training of modern ceramicists, of which there is currently a considerable shortage.

Subsidies must be provided primarily for basic research projects on materials with valuable technical properties, such as:

- Magnetic materials;
- Dielectric and ferroelectric materials, electric conductors, superconductors, ionic conductors, varistors, sensors;
- Materials with defined optical and electrooptical properties;
- Materials with outstanding mechanical properties such as great hardness, sturdiness, and resistance under static and dynamic stress at room temperature and high temperatures, wear resistance;
- Materials with particular thermal properties such as high resistance to thermal shocks, thermal conductivity, creep resistance;
- Materials with special chemical properties such as resistance to oxidation and corrosion.

The content of the program is divided into the following three topics:

- The chemical and physical basis of production processes;
- Constitution, structure, and properties;
- Trace analysis.

The objective of the program is to develop new processes and transfer laboratory processes to experimental production. New powders with a higher degree of purity and defined grain size and shape will be developed through targeted doping. Standard powders are to be produced at significantly lower prices. To this end, work was begun on developing a series of processes, as shown in Table 2.

New Powder Production Processes and Powder-Free Processes

[Text] In addition to the manufacturing processes, component properties largely depend on the properties of the ceramic powder from which they are made (Table 2).

Table 2: Overview of the Powder Production Processes Developed Within the Materials Research Program

Process	Oxide	Carbide	Nitride
Solid and solid-gas state reactions	Ion beam treatment (Al_2O_3 , ZrO_2)	Ion beam treatment (WC, B_4C)	Further development of powders from Si-nitriding (in particular, powder workup Si_3N_4)
Precipitation from the liquid phase	Hydrothermal synthesis (ZrO_2 , $\text{Al}_2\text{O}_3/\text{TiO}_2$) Reaction spray process (ZrO_2 , Al_2O_3) Sol-gel process (ZrO_2) Colloidally dispersed suspensions (Al_2O_3) Further development of ZrO_2 co-precipitated powders (separation) Improved Bayer Process (Al_2O_3) Flame hydrolysis (Al_2O_3 , ZrO_2) Plasma synthesis (Al_2O_3 , ZrO_2)	Polymer pyrolysis (SiC), further development of powders produced by the Acheson process (subsequent treatment (SiC)) Coat-Mix-Process (SiC)	Si-Diimide precipitation and crystallization (Si_3N_4)
Gas phase processes		Plasma synthesis (SiC)	Plasma synthesis (Si_3N_4 , AlN)

The reaction spray process used to develop multicomponent powders for the production of highly resistant oxide ceramics is also a process that holds out good economic prospects.

This process is based on the thermal dissolution or precipitation of solutions in a hot reaction space. An aqueous solution in which the components desired in powder are homogeneously dispersed is sprayed into the hot reaction tube through a jet. An agglomerate of powder is formed from each droplet through the spontaneous evaporation of the solvent, thus maintaining the homogeneous distribution of the selected components. The type of vaporization is determined by the raw materials and processing parameters used, and can result in a vast range of structures. The process is well-suited to high-speed, cost-effective powder production. In particular, it can be run as a continuous process.

The following composite materials, each offering potential applications, were manufactured as part of this project: ZrO_2 , $\text{Al}_2\text{O}_3/\text{ZrO}_2$, and $\text{ZrO}_2/\text{Al}_2\text{O}_3$.

The first strength measurements at room temperature were encouraging and gave the following results (sintering without pressure):

Material	Hardness MPa (N/mm ²)
ZrO_2	450-550
$\text{Al}_2\text{O}_3/\text{ZrO}_2$	700-800
$\text{ZrO}_2/\text{Al}_2\text{O}_3$	500-600
Pure Al_2O_3	450

The general goals in powder production are high purity powders and/or bringing the smallest possible grain sizes down into the submicron range. These objectives were met using various processes in a laboratory prototype. The results point to development potential which will be explored in greater detail in the future. Preference is given in the program to thermal precipitation from the liquid phase for the development of prealloyed multiphase powders with a homogeneous phase distribution—an example of a development of this type has been outlined. Current research is promising and must be followed through.

Materials and Process Development for Prototype Components

[Text] These are parts which, in terms of geometry and design load, represent a fairly broad range of similarly molded and loaded components. The goal of the work is to ensure that the potential shown by ceramic materials when single pieces are tested in the laboratory is fully maintained when the material goes into mass production at the industrial level.

Transferability is a particularly difficult problem for ceramics when voluminous and geometrically complex components are involved. Greater volume also makes for the accumulation of possible faults that cannot be tolerated by ceramics. Complex components require molding processes that guarantee the most homogeneous distribution possible for the powders, binders, sinter additives, pores, etc., as well as particular annealing and sintering processes to achieve compact structures with isotropic properties throughout the component.

The developments have been initiated on a broad base and draw on the following primary procedures:

- Selection of suitable materials;
- Selection of a process suitable for mass component production;
- Use of pure powders and procedures guaranteeing the avoidance of any further impurities.

The identification and documentation of as many influencing variables as possible and their interdependence is an interactive process that takes place at all stages of development. It frequently begins with several materials,

leads via component-like tests to the component, and takes into account testability and also production costs. To avoid increasing the amount of work required for a single component over the large number of possible ceramic components, all developments have been deliberately concentrated on "prototype components."

Since 1985, prototype components have been designed jointly with powder and ceramics producers and potential ceramics users. Table 3 provides an overview of materials and process development for components under the Materials Research Program. The "Exhaust Gas Turbosupercharger" project will be illustrated in greater detail as an example.

Table 3: Overview of Materials and Process Development for Components

Process Variables:					
	Oxide	SSiC	RBSiC (SiSiC)	SSN	RBSN (S,-(S)HIP-)
UP Sintering (uniaxial) [UP Pressen]			OP Sintering (quasiisostatic) [OP Pressen]		
SG Slip casting			DSG Pressure slip casting		
DF Pressure filtration			EA Electrophoretic precipitation		
SP Extrusion			SpG Injection molding		
Material Variables:			Oxide Al_2O_3 , ZrO_2		
SSiC Sintered silicon carbide			RBSiC Reaction sintered SiC		
SiSiC Silicon loaded SiC			SSN Sintered silicon nitride		
RBSN Reaction-bound silicon nitride			SRBSN Sintered reaction-bound silicon nitride		
HIPRBSN Hot isostatically sintered reaction-bound silicon nitride					
1. Static automobile gas turbine (GT) components					
GT intake spiral	DSG				
GT outer ring support			DSG	DSG	DSG
GT distributor [Leitgitter]			DSG	SG	SG
GT inner diffusor	QP				
GT inner ring	QP				
GT combustion chamber lining	QP				
2. Chargers					
Compression rotor	QP		QP		
ATL [expansion not provided] rotor	SpG		SpG		SpG
ATL intake spiral			SG		
3. Piston engines					
Valve guide	QP	QP			
Valve seat ring	QP	QP			
All-ceramic valve					SpG
Piston pin					SpG
Piston insulation (casing)	UP				SpG
Sheathed glow plug	QP				
Port liner	DSG			QP	
4. Energy technology					
Heat exchanger components			SG,SP		
Burner components			SG,SP		
Hot gas filter tube [Filterkerze]			QP		
5. Equipment and machine construction					
Spray nozzles			SpG		
Valve components	UP,QP				
Components for measuring equipment	UP,QP				
Components for HP Verd.				Sp	
2-Stroke engine (oil-free)				Sp	
Plate/tube	DF,EA				

The exhaust gas turbosupercharger rotor made of the ceramic materials silicon carbide (SiC) and silicon

nitride (Si_3N_4) is being developed in the project. Compared with metallic rotors, both materials have lower

weight and withstand very high temperatures. The project also addresses powder processing, injection molding and dewaxing, sintering, and machining stress, accompanied by non-destructive testing studies. The test program examines resistance to thermal shock, strength tests, and subsequent vehicle tests. The quality of the components is tested by nondestructive methods after each processing step.

The sintered and machined rotors were spun to fracture point on the centrifuge bench at 1030° C. With Si_3N_4 rotors, peripheral speeds greater than 500 m/s were reached. In the vehicle test a mass-produced metallic turbocharger was replaced by an SiC rotor. The rotor withstood 10,000 km at up to 96,000 rpm with a boost pressure of up to a maximum of 1 bar and exhaust temperatures up to 1030° C. The vehicle tests confirmed the expected rapid response of the ceramic turbocharger compared to the metallic mass-produced rotor, which was nearly three times as heavy. So far about 7,000 rotors have been manufactured by injection molding.

These results hold out good prospects of achieving the target of 500 m/s peripheral speed at 1230° C gas temperature and a failure probability of less than 0.1.

To exploit the advantages of the conventional slip casting process, such as a lower binder content and therefore fewer sources of error and easier annealing of large sections, high-pressure slip casting is being developed for the manufacture of hot-gas ducts for an automobile gas turbine from various Si_3N_4 variants. The goal is to reduce the currently excessive molding times and to make more complex component geometries accessible. After slip and mold optimization, components of varying sizes were nitrided and characterized with reference to degree of reaction, density, and structure. Parts produced out of sinter additive slip were then redensified by hot isostatic pressing, thus achieving 95-99.5 percent of their theoretical density. Bending strength test samples from HIPRBSN [hot isostatically sintered reaction-bound silicon nitride] components produce 4-point bending strengths of around 650 MPa. High pressure slip casting has good prospects for use in mass production as a fast and reproducible manufacturing process.

There are a series of processes that hold out prospects for achieving mechanical properties with a higher level of quality and greater reliability. However it has also been confirmed that the ability to produce voluminous and complexly formed components, subject to high dynamic and thermal stress, requires special developmental efforts. These efforts must therefore be the primary future concern of ceramics development.

Machining and Processing

[Text] The bonding of ceramics with ceramics and ceramics with metal is extremely important for processing. Three research institutes and seven companies are currently working on this task within the Materials Research Program.

The problem, particularly in cases where ceramics are bonded with metal, lies in their different structures, thermal conductivity, and expansivity. To an even greater extent the problem involves the limited ability of ceramics, as compared to metals, to avoid mechanical and/or thermal stress by plastic deformation. Without adequate practical mastery of bonding, ceramics as a structural material will be excluded from widespread application in industry. Since 1986, the following developments have been made in ceramics bonding: active soldering and brazing, soldering of precoatings, friction welding, and diffusion welding.

The development of active soldering methods for use at up to 800° C will be described by way of example. Silver-based soldering with titanium, copper, and/or indium additives were developed. These are good bonders for zirconium oxide with steel, gray cast iron, nickel-based alloys, and under argon and vacuum conditions (resistance of over 100 MPa). New solders with transversal resistance of 140-210 MPa were developed for silicon nitride materials as well. These solders can substantially reduce tensions in the soldered bonds as a result of their ductility and low melting point. Bonded components were successfully tested.

As a rule, final machining is essential in the production of ceramic components. Their resistance to machining and the danger of damage to the components are major problems in final machining. In view of the necessity of consistent, high-quality, and economic processing of ceramic components, the grinding and lapping processes are best suited to these materials.

Studies of selected grinding and lapping problem areas have demonstrated that efficient final machining to ensure good quality can only be achieved if both the machinery and tools used and the processing parameters applied are adapted to the properties of the ceramic material.

Plastics

[Text] Plastics have become extremely important not only in consumer goods—inconceivable now without the huge variety of synthetic products available—but also as materials for technology. The principal areas of application for these polymer materials are the automotive industry, mechanical engineering, equipment construction, and the electrical industry. The main properties required for these purposes are as follows:

- High strength, rigidity, and toughness combined with low weight;
- High thermal resistance over long periods;
- Good chemical resistance;
- Good electrical properties.

In addition to these areas of application, in which polymers are used as manufacturing materials, new fields of use have been opening up rapidly in recent years. These include:

- Electronics;

Communications and information technology;

Biotechnology.

In these fields, use is made of polymer properties which in the past were not commonly exploited, or were even unknown for these materials. Examples include high electrical conductivity or light conductivity. Among these "functional polymers" are: 1. Polymers with special electrical properties:

- Electrically conductive polymers;
- Piezoelectric polymers;
- Polymers with extremely low dielectric constants.

2. Polymers with special optical properties:

- Photoresists;
- Polymer light wave conductors;
- Polymers for optical data storage.

The development of plastics from raw materials for consumer goods into high quality manufacturing and functional materials is not only the result of applications-oriented polymer research but it also represents a challenge for such research.

Many achievements have been attained in basic research over the last few years resulting from interdisciplinary cooperation among chemical engineers, physicists, electronic engineers, and experts in other fields. This has provided considerable impetus to the development of new polymers. A deeper understanding of molecular structure, molecular interactions, and molecular architecture in polymers—achieved in part thanks to the potential offered by computer simulation and information and data processing—plays a key role here, as do new methods for defining the polymer structure. The Materials Research Program on "New Polymers" was initiated to support research in German industry and institutes, keep pace with the enormous speed of international developments in polymer research, and maintain a leading position. The research concentrated on the following R&D topics:

- Polymers with extreme stability under stress;
- Polymer alloys and copolymers;

- Polymers with special properties, e.g., electrical, electronic, magnetic, and optical characteristics;
- Polymers as information carriers.

While structural polymers had greater weight at the beginning of the program, consuming more than 50 percent of the project resources, there is an unmistakable trend towards assigning greater importance to functional polymers. This is only right, and stems from the internationally held view that functional polymers have particularly high innovative potential and that a major breakthrough in this field is imminent.

Polymers with Extreme Stability Under Stress

[Text] Extreme stability under stress may cover a wide variety of demands made upon the material, in particular, high thermal resistance and strength. Measured against their theoretical values, only a fraction of the strength of polymers has been utilized to date. Of course, the theoretical values cannot be fully achieved, but there is nevertheless an immense development potential here for high-strength polymer materials. Polymer research is working on reconciling extreme stability under stress with adequate processibility of the material into semifinished products and components. The principle set down in the Materials Research Program is of particular relevance here: "Equal priority will be given to developments in the composition of a material and its processing technology."

The program's approaches for achieving extreme stability under stress include the targeted construction or development of a molecular architecture with defined molecular chain lengths and specified side-chain structure, internal molecular strengthening in polymers, and the synthesis of liquid-crystal polymers (LC polymers).

LC polymers are characterized by rigid, largely aromatic sequences of molecules, which must be of a minimum size. These macromolecules tend to adopt an extended position and can be aligned in electrical or magnetic fields. This makes it possible to influence the degree and the direction of orientation. This targeted anisotropy provides high strength and rigidity, and greatly improves thermoforming resistances. Table 4 outlines the properties of two LC polyesters developed under the Materials Research Program. To highlight the vast improvement in properties offered by these materials, values for a "classical" mass synthetic material, polyethylene terephthalate (PETP), are provided as a comparison.

Table 4: Comparison of Properties Between LC Polyesters and Polyethylene Terephthalate

Property	Unit	Polyethylene terephthalate	LC Polyester 1	Newly-developed LC Polyester 2
Tensile strength	MPa	73	170	220
Impact strength	Kj/m	1-4	65	58
Ester number modulus	GPa	2.85	18	22
Flexural strength	MPa	118	160	180
Thermoforming resistance HDT [high pressure temperature]	°C	70-80	250	140

The focus of R&D work on LC polymers is shifting increasingly toward the development of processing techniques for these materials. The directionality of the properties, strongly influenced by processing into a preform, sets processing engineers and preform designers the task of producing as much anisotropy as necessary during, for example, injection molding or extrusion (increased level of properties) without sacrificing too much isotropy so that various stresses can be withstood.

Selected prototype products made of these LC polyesters are currently being produced on a semicommercial scale and their characteristics comprehensively documented.

Polymer Alloys and Ordered Copolymers

[Text] The blending and alloying of various raw materials has always played an important part in the history of the development of materials. In metallurgy, alloying skill remains the most important method of tailoring properties or combinations of properties for metallic materials. For some time now the plastics industry has also been attempting to combine polymers with quite different properties into materials created out of the sum of the properties of the individual components, which are either difficult or impossible to create by other means. For example, the alloying of hard and brittle thermoplastics with soft but tough rubbers results in "impact-resistant" synthetics, different variations of which have since been opening new fields of application. Property combinations of this sort may be attempted either by creating polymer alloys, or by synthesizing ordered copolymers, or by a combination of these two approaches.

The objective here, as in the case of the LC polymers, is to achieve maximum improvement in one or several desired properties without excess detriment to the other properties. "Pole diagrams" provide a qualitative overview of the properties achieved. Figure 5 shows a pole diagram for a polyphenylene ether and polyamide-based polymer alloy developed under the Materials Research Program.

Figure 5: Pole Diagram of a Polyphenylene Ether and Polyamide Polymer Alloy [not reproduced] The example shows that with this alloy the favorable properties of polyphenylene ether—low water absorption, good dimensional stability, low distortion, high impact resistance and high rigidity—can be combined with those of polyamide—low stress cracking susceptibility, good resistance to solvents, easy processing, and good thermo-forming resistance. This opens a large number of potential applications: bodywork parts such as engine hoods and trunk lids, heavy-duty housings for the automobile and electrical industries, etc.

The successful example illustrated here may not, however, disguise the fact that a large number of potential new material properties are waiting to be opened by research and development of polymer alloys. In particular, understanding the thermodynamics of alloy formation holds the key to more systematic alloy development.

It is often impossible to achieve complete miscibility of different polymers, in most cases for thermodynamic reasons. In practice, two-phase or multiphase systems tend to occur that are frequently incompatible, or only partially compatible, with one another. At worst, this can result in totally unusable materials. The decisive influence on the properties of polymer alloys is phase morphology which, if controlled and adjusted according to requirements, is the determining factor in phase compatibility and hence the key to the creation of applications-oriented properties for new, high-performance polymer alloys. Phase compatibility of this sort can, for example, be achieved by synthesizing suitable homopolymers and copolymers for use as components in binary polymer alloys; another method is controlled physical or reactive alloy technology. A particularly interesting category of polymer alloys which are just beginning to be developed are the physically- or chemically-linked interpenetrating networks (IPN). However chemically-linked IPN's cannot be processed thermoplastically at higher levels of cross-linking.

In a project started at the end of 1984, extensive tests were conducted on thermodynamic mixed behavior and phase compatibility in polymer alloys obtained from homopolymers and copolymers, and the phase morphology characteristics were documented. The model system, polymethyl methacrylate/polystyrol, shows all conceivable degrees of compatibility, according to the proportions making up the alloy, from homogeneous compatibility to marked incompatibility. Even the markedly incompatible blends could be forced into a homogeneous state. Below glass temperature, this forced homogeneous state proved stable.

A new type of reaction extruder for the creation of stable, multiphase polymer alloys by means of reactive alloying has been developed and operated since May 1986. New types of polymer alloys with strong physical interactions have been synthesized. Extensive studies have resulted in a large quantity of basic data on phase compatibility, physical phase interaction, and the working of newly-synthesized polymer compatibility mediators. Temperature tests provided proof of the phase stability of systems created by reactive alloying.

Properties of ordered copolymers, which are of interest both in their own right and as components in alloys, can also be combined. In this case, the property profile stems from the chemical linking of various monomers. The factors that determine the property profile for ordered copolymers are molecular structure, molecular sequence, and molecular architecture. The ability to create alternate or block copolymers in reproducible form requires a detailed knowledge of the kinetics and thermodynamics of polymerization. The work schedules for projects on this subtopic are structured accordingly.

The results achieved to date in the R&D work in progress have yielded important basic knowledge about the reaction kinetics of the polymerization processes used to

manufacture copolymers. For example, in a project started in July 1984 thermoplastically processible crystalline polymers based on neopentyl glycocarbonate (NPC) were synthesized and have already moved out of the laboratory into semi-industrial scale production. The thermoplastically processible crystalline material is distinguished by high resistance to solvents and is outstanding for melt spinning into fibers.

Polymers With Special Electrical, Electronic, Magnetic, and Optical Properties

[Text] In general, synthetics rank as electrical nonconductors, and these insulating properties are further optimized for specialized fields of application in electrical engineering and electronics. Another line of development is moving in precisely the opposite direction, aiming to endow synthetics with high (intrinsic) conductivity, and to do so without the admixture of electrically conductive particles. Here again, the R&D work is based on knowledge gained from basic experimental and abstract research into the physics and chemistry of the molecular structure of polymers. Since the discovery of the first crystalline organic compounds, which conduct electrical current in the same way as metals, there has been a flood of developments. Functional polymers with electrical conductivity comparable to that of metals are already state-of-the-art. Table 5 provides an overview of the conductivity and density of various materials, as available in 1985.

Table 5: Conductivity and Density of Various Materials
(Source: UMSCHAU Volume 11, 1985)

Material	Conductivity (S/cm)	Density (g/cm ³)
Copper	6×10^5	8.9
Mercury	10^4	13.5
Iodine-doped polyacetylene	10^3	0.8
Phenyl sulphomate-doped polypyrol	1.5×10^2	1.3
Carbon black-reinforced polymers	10^{-1}	1.0
Uncomplexed polyacetylene	10^{-7}	0.5
Polystyrol	10^{-16}	1.05

Some years ago in Japan, Shirakawa reported iodine-doped polyacetylene with considerable intrinsic conductivity. FRG research teams also succeeded in producing conductive polyacetylene, but at first all attempts failed to render the electrical conductivity produced by doping polyacetylene with iodine that is thermally stable and insensitive to oxygen and water.

The Materials Research Program has placed particular emphasis on electrically conductive polymers, to which it has devoted numerous R&D projects. Enormous R&D efforts are being focused on acquiring basic knowledge of the physics of electrical conductivity in polymers and the

chemical synthesis of conductive polymers, so that new polymer materials with high electrical conductivity may be synthesized and their conductivity stabilized beyond the processing stage.

One possible approach has been under study since 1984, the goal being the synthesis of conductive polymers displaying high crystallinity without chemical cross-linking points. These polymers should only have such amorphous areas as permit targeted doping, but which contain no defective points or specific groupings such as allyl functions with particular oxygen sensitivity. Whereas polyacetylene film with conductivity of about 10^3 S/cm is being produced by Shirakawa's process, polyacetylene with conductivity greater than 10^5 S/cm has also been produced within this project. This means that for the first time, the "specific conductivity" of copper and silver, that is, the conductivity specific to density, has clearly been exceeded.

One of the first successful applications is emerging for polypyrol film about 300μ thick, which have been developed with a long-term stable conductivity of up to 200 S/cm. These films have been used as electrode materials in batteries.

These batteries have a higher storage capacity, are heavy, and metal-free. Another advantage is that they can be flexibly configured, thus permitting new structural solutions for electrical equipment.

Another line of development, started in September 1985, used electrochemical polymerization to synthesize various electrically conductive polymer materials with electrical conductivity of about 10^{-2} S/cm. The products included conductive transparent films with surface resistances in the 10^8 - 10^{11} Ohm range, suitable, for example, for permanent antistatic finishing on packaging materials.

The development of highly translucent glass in the sixties set in motion rapid developments in optoelectronics and beam waveguides which have resulted in completely new communications opportunities and an enormous increase in information exchange capacity. Although until the early eighties glass fibers were practically the only beam waveguides to be considered because of their low light attenuation new polymer materials are now penetrating certain areas of this technology. Polymers with low attenuation are now so far developed that they can replace quartz glass beam waveguides in short-range communications. Although the level of attenuation which can currently be achieved in polymer beam waveguides such as polymethyl methacrylate (PMMA) is still several times greater than that of glass fibers, an expanding market is nonetheless emerging on account of interesting advantages in processing and system costs. Table 6 shows characteristic values for beam waveguides.

The light attenuation of PMMA can be favorably reduced and the values given in Table 6 further

improved by targeted deuteration or halogenation. However, the material's processibility decreases rapidly at high levels of halogenation. It is precisely this processibility—in this case, the spinning of the polymer into optical fibers—which is an indispensable prerequisite for the technical utilization of materials such as these. In addition to high translucency, the material should also present a high glass transition temperature and good bonding to the core-cladding interface.

Table 6: Characteristic Values for Beam Waveguides
(Source: CHEM. ING. TECHNIK Volume 59, No 8, 1987)

Fiber type	Data transmission distance (without intermediate amplifier)	Application field
Polymer fiber (PMMA)	0.15 km	Short-range communications, e.g., production automation
Polymer fiber (per-deuterized PMMA)	1.5 km	Computer networks, local network field
Glass fiber	30 km	Telecommunications, computer networks, local networks

In a project started in early 1985, 60 monomers containing fluorine and chlorine have been synthesized to date, and their homopolymerization and copolymerization behavior has been studied. The special clean room technology indispensable for this was elaborated and optimized. A newly-designed prototype spinning unit made it possible to produce polymer beam waveguides with attenuation values of less than 200 dB/km (glass: 1 dB/km). It was proved that surprisingly high bit rates of about 1 GBit/s at 24 m could be achieved with polymer beam waveguides. Calculations have shown that optimal combinations of core and cladding polymers can be achieved for bicomponent fibers.

Since January 1986, six research groups—three at chemical companies and three at research institutes—have been trying to break into a completely new dimension for functional polymers, "ultrathin polymer films." "Ultrathin polymer films" are, for example, polymer films in the submicron range produced by the Langmuir-Blodgett method and made up of only a very few layers of molecules. Such ultrathin organic films with a controlled molecular structure are expected to have completely new physical properties (for example, electrical, electronic, ionic, or optical) with many potential uses. Extensive work on synthesizing new types of monomers or polymers suitable for the Langmuir-Blodgett technique has begun, and a start has been made on fundamental research into various physical effects and on characterizing the mechanical, electrical, and optical properties of mono- and multilayers, taking special account of their suitability for nonlinear optics. Results

thus far are extremely promising. Numerous new types of monomers resulting in films with outstanding thermal and chemical stability have been synthesized. Films with thermal stability up to 270°C have already been successfully produced from suitable polymers. In the case of functional amphiphilic polymers with hydrophilous head groups, which are being considered for possible application in nonlinear optics, nonlinear optical effects of the second order have been recorded that are greater than those achieved to date.

Polymers as Information Carriers

[Text] Worldwide technological developments in the nineties could well be determined by progress in microelectronics, especially in data storage and processing. Progress in this area is directly linked to the capability to store the greatest quantity of data in the smallest possible space. A major contribution to the development of advanced memories could come from polymer materials. For polymer materials to be successfully developed as information carriers, close cooperation is necessary between the chemical industry—for the synthesis and characterization of new materials—and the electrical engineering and electronics industry for the relevant developments in systems and components. Optomechanical memories, which are particularly cost-effective per bit and storage density, can currently achieve packing densities of about 10^8 bit/cm². Packing densities of up to 10^6 bit/cm² have been achieved in two-dimensional storage with photopolymers. The limits that can be achieved with molecular memories are determined by quantum and tunnel effects, the electron path lengths and the electrical field strength. A molecular memory could be created by using electron beams to exert a targeted influence on individual molecules or molecular bundles. At the moment, however, the limited resolution capacity of electron beams still precludes molecular orientation at the individual molecule level. Despite this, an increase in storage density from 10^{11} bit/cm² as compared with conventional optomechanical memories could nevertheless be achieved.

Requirements thus include the development of polymer materials with high sensitivity to electron radiation, good resolution, good bonding to base materials, and suitability for dry etching processes. Technical solutions to these requirements cannot, however, be expected in the short term, since intensive research is still needed to provide essential basic knowledge. The preliminary results include ultrathin polymer films and special liquid crystal polymers (LC polymers) with reversibly modifiable phases.

In mid-1986, R&D work was begun on the synthesis of various liquid crystal side-chain polymers in the form of homopolymers, copolymers, and block polymers, and on examining them for phase-orientability and reorientability in the presence of external electrical and magnetic fields. Results of importance for data storage are also expected from the research on ultrathin films. Marked

improvements have been made in the resolution, sensitivity, and contrast of LC polymer films for optical storage. The intention is to fund projects on "Polymers for Information Storage" within the Materials Research Program in the coming years.

Total Costs and the BMFT share of projects on new polymers started by 30 June 1988: Total Costs: DM199.6 million BMFT share: DM103.3 million

Distribution of Resources for R&D Topics

Polymers with extreme stability under stress	28%
Polymer alloys and copolymers	17%
Polymers with special properties, e.g., electrical, electronic, magnetic, and optical characteristics	53%
Polymers for information storage	2%

Functional Ceramics

[Text] Functional materials were the major element of the polymers subject area at the start of the Materials Research Program. However, since ceramic materials in particular still show very great potential for the development of materials with special physical properties, such studies will be pursued more intensively in the future. Emerging fields of application are electronics, data storage and data processing, communications and energy technology, metrology, and control engineering. The funding will complement the DFG's priority program with more application-specific projects. A project started in early 1988 for the production of close tolerance dielectric ceramics for microwave components and four-stage implementation is an example. In energy technology, a project seeking a more economic and reproducible production method for solid electrolytes for sodium/sulfur batteries stands out.

Total costs and the BMFT share of the ceramics projects started by 30 June 1988: Total costs: DM251.0 million BMFT share: DM124.4 million

Distribution of Resources for R&D Topics

Basic materials and process development	14%
DFG priority program	15%
New powder production and powder-free processes	12%
Materials and process development for prototype components	53%
Machining and processing	6%
Functional ceramics	under preparation

Powder Metallurgy

[Text] Powder metallurgy as a manufacturing process for metallic components has two-fold importance for materials research.

First of all, this manufacturing process—the production and subsequent compression of metallic powders into

semifinished goods and components—makes it possible to produce small, geometrically complex components approaching the final contour with a very economical use of raw materials and little finishing. This economic aspect has resulted in parts manufactured by powder metallurgy—mainly out of iron-based alloys—being produced and used in large numbers.

As for materials development, powder metallurgy's particularly high innovative content lies in the use of the high cooling speeds achieved in powder production and in the special densification processes employed. These combine to produce alloys that cannot be obtained from melting processes and which present the desired new properties. The powder metallurgy projects therefore focus on the basic development of new powder metallurgy processes to improve and further develop more economical materials such as aluminum- and iron-nickel-based alloys. R&D projects designed to create particularly cost-effective component manufacturing processes complement these lines of development.

New Powder Production Methods

[Text] Rapid quenching of an alloy cast at temperatures at which no further atom migration processes can take place allows the production of alloys whose properties differ—in part drastically—from equivalent equilibrium phases. This provides an opportunity to produce materials with totally new or much-improved properties. Well-known examples of this are the "metallic glasses," which are widely applied in electrical engineering because of their particular magnetic properties.

To produce the fastest possible cooling rates, which are necessary to obtain new material properties, the materials must first be produced as powders or thin strips. The powder particle sizes diminish as the cooling-off rates increase, and lie between 60 micrometers and a few nanometers. A feature common to all processes is that the molten particles have to be brought into contact with a very effective heatsink such as, for example, a water or gas jet.

Various processes of water, gas, or rotary atomization of casts are already part of the current state of the art. However the cooling rates, the purity of the powders, the particle sizes, and their distribution are often unsatisfactory from the technological and/or economic point of view. The program's objective was therefore to develop new, nonconventional powder production processes. Three teams, each drawn from an institute and several industrial firms, started on the development of new powder production processes in 1986.

The first development was devoted to the contact-free ultrasonic standing wave atomization. This process is based on the disintegration of a liquid jet in the pressure nodes of a high-intensity, ultrasonic standing wave in a gaseous medium. Initial atomizing experiments on molten metal between two ultrasonic converters confirm

that sufficiently fine atomization can be achieved, provided a minimal overpressure of less than 5 bar is maintained. An atomization plant is currently being modified and the feasibility of metallic powder production is being analyzed.

The second development is aimed at atomization using liquified gas. Liquid nitrogen is used as the atomization medium. The first experimental tests were carried out after a prototype had been constructed. In the case of eutectic PbSn and CuPb alloys, they yielded fine powders whose particle size distribution depended on the atomization parameters.

The third line of development is laser beam atomization. A pulsed NdYAG laser demonstrated in initial experimental tests that the tested metals, alloys, and metallic glasses could be converted into the finest powders. The particle size spectrum extends down to a few tens of nm. Oxide ceramics can also be produced in this manner. Not least to achieve a high yield, plant and processes must be optimized and powder morphology, phase composition, and powder properties must be further studied.

An additional line of development aimed at the production and characterization of metals with nanocrystalline structures (crystallite size about 10nm) is of fundamental importance. This work, started at an institute in 1986 and in a company in 1987, has provided the following results so far: the interfaces surrounding the crystallite show a broad distribution of interatomic intervals comparable to a gaseous structure, but not to any hitherto known amorphous or crystalline structures. The particular structure of these interfaces is held responsible both for diffusion-controlled improvements in properties (e.g., electric properties), and for the particular deformation stress properties of normally brittle materials (e.g., CaF₂ and TiO₂). However it is still early yet as far as both comprehending and producing these materials through various powder metallurgy processes are concerned. As new or much-improved properties are emerging for both metallic and ceramic materials, the Materials Research Program will devote particular attention to nanocrystalline materials in the future.

Development of Improved Aluminum- and Titanium-Based Light Metal Alloys

[Text] Light alloys, in particular aluminum alloys, are firmly established in numerous technical applications owing to their good weight to strength ratio. Their range of applications are restricted primarily by the loss of strength which occurs even at relatively low temperatures (Al alloys at 200° C, Ti alloys at 400° C). The aim was therefore to exploit the potential of powder metallurgy to obtain a significant increase in working temperatures and strength while maintaining low density, low thermal expansion coefficients, or high corrosion resistance, according to requirements.

Powder metallurgy opens two roads toward the stated goals: a) The rapid solidification (atomization) of Al or Ti materials with alloyed compounds that could not have been achieved by smelting; b) The grinding of oxides and carbides by mechanical alloying or reaction grinding [Reaktionsmahlen] (dispersion hardening).

Both approaches are adopted in the joint projects underway in this area. While the work begun in 1987 on developing new titanium alloys for applications at higher temperatures (650 - 700° C) is still in the early stages, the joint projects that have been working since 1985 on creating new aluminum alloys show remarkable interim results:

- An Al-9Zn-3Mg-1, 5Cu-X-based alloy type has been developed for maximum strength and reaches an apparent yielding point of 750 MPa and a fatigue limit of 300 MPa. Intended applications: aerospace, machine construction.
- A second alloy type aims for both high strength and good resistance to corrosion. An apparent yield point of 470 MPa and a fatigue limit of 240 MPa were achieved with an Al-4Mg-X base. Intended applications: machine parts exposed to damage by corrosion.
- An Al-Si20-X-based alloy was developed which at 150° C shows both a vibration fatigue strength of 190 MPa and a low thermal expansion coefficient of 15 x 10⁻⁶ K⁻¹. Intended application: automobile connecting rods.
- An alloy was developed in the Al-Mn10-X-Y system which at 300° C shows an apparent yield point of 230 MPa and a resistance to vibrations of 90 MPa. Intended application: compressor wheel for aircraft turbines.

These interim results demonstrate that German research and development work on aluminum alloys produced by powder metallurgy has regained a leading international position in a short period.

Development of New Processing Methods

[Text] The successful application of newly-developed materials requires that the methods used to process them be developed at the same time. This is a common, and successful, practice in the joint powder metallurgy projects. Component manufacturers and users normally take part in the projects as partners.

In addition to making it possible to process new materials, however, powder metallurgy also provides a very rational way of processing conventional materials into components. The successful development of "spray compacting," a new manufacturing process for sheet steel, tubes, and profiles, is an example of this approach.

The atomized melt in the form of droplets coats the substrate directly until the desired thickness is achieved.

Powder production, compacting, and shaping (for semi-finished goods) are thus combined in a single processing step.

The first results of this development clearly demonstrate the great development potential of this method. It is already known that both flat products and tubes, round billets, and small parts can be manufactured from both steel and multilayer materials.

Total costs and the BMFT share of powder metallurgy projects started by 30 June 1988: Total costs: DM93.7 million BMFT share: DM57.6 million

Distribution of Resources for R&D Topics

New powder production and processing methods	35%
Al-, Ti-, (Mg-) based alloys	27%
Cu-, Fe, Ni-based alloys and mechanically resistant materials	33%
Materials with graded structure	5%

Metallic High Temperature and Special Materials

[Text] Metallic materials continue to be indispensable in many areas of technology. They have high strength and toughness, good thermal conductivity, and can be produced and processed economically. The application of new findings in metallurgy, casting, solidification (e.g., rapid solidification), and further treatment has improved their strength, toughness, and high temperature and corrosion-resistance, thus demonstrating that metals produced by smelting still have additional development potential. A factor that limits the application potential of metallic materials is heat resistance. The line of development being followed, however, is to run technical processes at higher temperatures, thus improving efficiency or product properties and reducing production costs. The Materials Research Program singles out the development of metallic high temperature materials for hitherto inaccessible temperature ranges as a priority topic.

Material	Operating Temperature (° C)		
	State of the art in 1985	Materials Research	Goal
Superalloys	1050	1100	
Ferrite steels	565	650	
Ti alloys	400	650	
Al alloys	200	450	
Intermetallic phases	<1000	1200	

In many cases it makes economic sense and is technologically advantageous to demand special properties of components only on the surface or in the uppermost material layer, where they can be incorporated by means of an appropriate treatment. In this way protective layers can be built up against high temperature damage, abrasion, or corrosion without adversely affecting the structural properties of components.

High temperature property improvement is not the only task with many metallic materials; other aspects such as reducing the weight of the components or raising corrosion resistance must also be considered. Materials with special properties of this type are described as "special materials."

All questions affecting reliability, durability, material characterization and testing, and the links between them are also important. The Materials Research Program places special emphasis on further development in these areas.

Figure 3: Intermetallic Phases as High Temperature Materials, Source: Max Planck Institute of Iron Research [not reproduced]

Intermetallic Phases as High Temperature Materials (Figure 4)

[Text] This group of materials with strong metallic atomic bonds can be used to develop new materials which are stronger than metal alloys and tougher than ceramics, even at high temperatures. Phases for applications as lightweight materials at operating temperatures under 1000° C and for applications above 1000° C are important here. Titanium aluminides are among the lightweight materials already examined in detail. Nickel aluminides with thermal resistance comparable to conventional high temperature alloys are also well-known. Titanium aluminides and nickel aluminides are not suited to operating temperatures between those of superalloys and ceramics. Other systems that have not been studied much to date must be considered for this purpose. In this connection the following phases were measured for thermal resistance: (Co, Fe) Al, NiAl-Cr, NbAl₃, Co₂TiAl, TiCr₂, Ti₅Si₃, TaFeAl.

The results compare with known phases as follows:

Material	0.2% offset yield at 800° C	(MN/m ²) at 1200° C
UA 6000 superalloy	710	110
Known intermetallic phases		
Advanced Ni ₃ Al	870	
Ni ₃ AlC	540	
(Co,Fe) ₃ V	420	
TiAl-Nb	370	
NiAl	130	
New intermetallic phases		
(Co,Fe)Al	420	52
NiAl-Cr	410	150
NbAl ₃	>800	160
Co ₂ TiAl	700	170
TiCr ₂	>1200	280
Ti ₅ Si ₃	>1200	430
TaFeAl	>1200	820

Further Developed Superalloys

[Text] The demand for extremely tough materials for applications at over 950° C, for example in turbines,

engines, and heat exchangers in power stations is constantly increasing, so work on further improving thermal resistance, tensile strength, and corrosion resistance is of great technical importance. Thirteen firms and five research institutes are working jointly on developing this class of materials within the Materials Research Program. Their priority concern is the introduction of a high-strength, light monocrystal alloy for turbine blades. This will contribute to the development of components in nickel-based metallic high temperature materials with considerably improved operating properties, including increased high temperature stability, corrosion resistance, thermal fatigue stability, and operating temperature. The work is very broadly-based, involving the development of suitable casting parameters and their correlation with the respective structural parameters, guarantees of reproducibility and the correlation of structural parameters with properties relevant to use in engines. In the first 1 and 1/2 years of the 4-year term, casting tests and the determination of the basic properties of the selected alloys confirmed that they could be used. Structural parameters were defined and methods for measuring them were established. The cost-effective manufacture of nickel-based mono-crystalline turbine blades has not been perfected in the FRG. Since know-how cannot be transferred from the leading countries in this field, the United States and the UK, the work begun under the Materials Research Program will serve to catch up with international developments. The partial results achieved so far indicate that this purpose will be fulfilled.

New Lightweight Metal Materials

[Text] Work on lightweight metal alloys covering a range of widely differing aspects is currently being carried out as part of the Materials Research Program.

There are three main topics:

- Development of casting technology for the manufacture of lightweight metal structural parts for aviation and transport. Here large-format, integral castings with high mechanical strength will be manufactured from new lightweight metal alloys by low-pressure sand-casting and low-pressure high quality casting.
- Bonding technology both for high strength aluminum alloys with a view to superplastic transformation and for aluminum-lithium alloys.
- Development of aluminum-lithium alloys with a Li content of up to 10%.

The low-pressure high quality casting project has so far yielded extremely positive results. Once melting temperature and mold temperature have been mastered and the influences exerted by particle size and finishing are known, part of the complexity of the wing flap suspension can be mass produced with the required high mechanical parameters and X-ray quality control. These findings made it possible to manufacture a large part such as an aircraft cargo hold door (measurements: 1.3 x 1.0 x 0.2 m) within the required limits.

Advanced Ferritic Materials

[Text] The goal of this joint project is the development of new high temperature weldable ferritic steels as well as special materials to withstand complex stresses.

The efficiency of fossil-fueled power plants is currently limited by the stress stability of the high temperature weldable ferritic steels in use. An attempt is therefore being made to raise their operating temperature from 550 to 600° C or even higher. Apart from higher costs, the use of known high temperature austenitic steels would involve the disadvantages of greater thermal expansion, lower thermal conductivity, worse cutting properties, and in some cases increased susceptibility to stress corrosion cracking. For this reason high temperature ferritic steels for application at higher temperatures require further development.

One possibility lies in increasing the nitrogen pressure content in thermal resistant ferritic steels. New technologies now make it possible to increase nitrogen content far beyond the degree of solubility reached at atmospheric pressure. By increasing the nitrogen pressure content during electroslag remelting, the elastic limits and long-time creep strength of austenitic steels with nitrogen contents of up to 1 percent were increased and good levels of toughness were retained.

With regard to the subject of this research project, ferritic steels with increased nitrogen pressure content, the first positive results are emerging. For example, at 550° C the toughness of the standard 13 CrMo 44 steel increases from 516 to 563 N/mm² when the nitrogen content increases from 0.09 to 0.15 percent.

Characterization and Testing, Reliability, and Durability of Metallic Materials

[Text] Matters concerning the characterization and testing, durability, and reliability of materials play a decisive role in the development of new materials and the assessment of their subsequent uses. Where particular processes cannot be applied to new materials, they have to be adapted or new processes must be developed.

The following two lines of development are examples of this:

In the development of a structure-dependent material model for complex high temperature stresses, the assessment of safeguards against failure for components subject to complex high temperature stresses will be studied. New methods will be worked out so that the properties of new high temperature-resistant materials with a defined reliability (defined failure probability) may be better exploited. These methods will be tested on turbine rotors and cooled gas turbine buckets. Preliminary tests to examine various new methods of measurement are underway and some of the results are promising. In the case of the turbine rotors, the quantitative description of

the prevailing variables is so far advanced that it can be assessed against the results of LCF [expansion unknown] tests. The turbine bucket assessment draws on a materials database. With the project one-quarter completed, no final conclusion can yet be drawn as to whether all the studies initiated will lead to success; however the preliminary tests are promising. The approach adopted here in the development of a failure reliability model for components subject to complex high temperature stresses is extraordinarily difficult, but the combination of metallography and materials mechanics with stress histograms is also highly innovative. The work is very topical and has already attracted international attention.

There is increasing interest in employing neutron radiography as a method of nondestructive materials testing on a wide industrial basis. An extensive assessment of the efficiency of neutron irradiation methods in nondestructive materials testing is being made within the joint project by examining several hundred samples with defined faults from aerospace technology, electrotechnical and electronic engineering, and mechanical and civil engineering using neutron radiography or neutron computer tomography and, by way of comparison, with conventional nondestructive materials testing methods. Neutron irradiation methods provided very good results, particularly regarding the detection of corrosion, water voids in honeycomb structures, cracking and delamination in electronic components, faults in ceramics and fiber composite materials, ceramic residues in turbine buckets, and wrongly inserted seals between metals. The potential of digital image enhancement and image interpretation processes in neutron radiography was successfully demonstrated. The industrial use of neutron irradiation methods is made possible by new and existing neutron radiography facilities.

Protective Coatings

[Text] The development of protective coatings designed to protect components against high temperatures, corrosion, and abrasion is, apart from process development and optimization, primarily a matter of materials development.

One joint project is using vacuum plasma spraying (VPS) to produce refractory nitride, carbon nitride, and boride-based antiabrasion coatings which, like the well-known tungsten carbide protective coatings, can be used in conventional mechanical engineering. From the operative point of view the VPS process provides suitable conditions for processing reactive wettable powders. Various wettable powder grains of the following spraying materials were examined: ZrN, TiN, TiCN, TiC, TiB₂, CrB, and CrB₂.

The right spraying parameters were determined for processing, the layers were examined metallographically, and coating properties such as porosity, hardness, and abrasion resistance were ascertained. The ductility and bonding of the coating properties can be substantially

improved by the addition of a metallic binder such as pure titanium without compromising wear resistance. Two-stage factor analysis was therefore used to optimize the mechanically resistant material content in the Ti + TiN + TiC + TiB₂ system with regard to the type and quantity of the mechanically resistant ingredients.

The results achieved to date derive mainly from laboratory experiments. They give grounds for confidence that the properties of conventional coatings can be improved. This emerges particularly in high temperature coatings with increased resistance to oxidation and abrasion. In view of the great application potential for protective coatings, and the research work underway worldwide, German activities are likely to be intensified. Some basic work must still be followed up.

Total costs and the BMFT share of the metallic high temperature and special materials projects started before 30 June 1988: Total costs: DM176.0 million BMFT share: DM99.5 million

Distribution of Resources for R&D Topics

Intermetallic phases as high temperature materials	15%
New light metal materials	10%
Further developed superalloys	19%
Advanced ferritic materials	10%
Characterization and testing, reliability, and durability of metallic materials	35%
Protective coatings	7%
Other	4%

Composite Materials

[Text] Nature shows man in a multitude of ways how a minimum of material can be used to make structures which are highly stable and at the same time very light. Such requirements can be met by composites, which are in a way both the oldest and the most modern materials. Examples of the oldest materials are wood or bone. But composites are also some of the most modern materials, without which many engineering achievements in aeronautics and astronautics, electronics, mechanical engineering involving fast-moving parts, consumer goods, and the leisure field would simply not be feasible. The crucial technological significance of composites, which will certainly continue to grow, is based on the principle formulated by G. Slayter that a composite, constituted for example from fibers or particles of one material embedded in a matrix of a different material, has properties which neither of the two components possess on their own.

Modern high performance fibers combined in suitable matrices, for example carbon or aromatic polyamide fibers—stronger than steel and lighter than aluminum—open prospects for technological applications which so far have been regarded as hardly practicable. In addition to the advantages of high stability and rigidity in conjunction with low specific gravity, they are also resistant

to material fatigue and have good flow stress properties, low thermal expansion during temperature fluctuations, as well as good chemical and corrosion resistance in composite plastics.

Because of these obvious advantages, composite materials have become established in many high technology sectors such as aerospace—in some instances replacing traditional materials such as metal. These are primarily areas of application where the advantages accruing from the technological properties of composites at least offset the relatively high cost of producing them, and where only a small number of pieces are produced. The excessively high production costs of polymer composites frequently prevent their wider use. In the case of metal matrix and ceramic matrix composites, there is also a considerable lack of basic knowledge of physical and chemical properties and processing techniques.

As a result of this situation, materials research activities are focused in the following areas:

- Composite materials with polymer matrices;
- Composite materials with metallic matrices;
- Composite materials with ceramic matrices;
- New and improved reinforcing fibers;
- New composite structures.

Composite Materials with Polymer Matrices

[Text] The technological application of composites with polymer (plastic) matrices will increase when components with the highest possible integrated functionality can be produced cheaply and quickly. It must also be possible to conduct nondestructive quality testing during

production and operation. Yet there are still problems of "classical" materials development to be solved for a great many possible applications: the synthesis and production of suitable polymer matrix systems with increased thermal resistance, matrices that can be thermoplastically treated, and matrices with properties optimized in relation to the ratings of the reinforcing fibers (strength, expansion, interfacial bonding).

Polymer matrix materials are usually reinforced with fibers (short or long fibers). Duroplastics such as epoxy, phenol, and polyester resins are primarily used at present to make high performance glass, carbon, or aramid fiber-reinforced composites. These resins allow operating temperatures up to 180° C. (Figure 6: Structure of Fiber Composites with Polymer Matrix [not reproduced])

Higher operating temperatures are appropriate for duroplastics, which is a challenge for processing and manufacturing engineering in particular. The components are manufactured both manually and mechanically by impregnating fibers with resin and subsequently molding them in dies, often under pressure and at high temperatures. The part cannot be further shaped after hardening. This is the advantage of thermoplastic matrices: semi-finished products can be processed into components anytime, anywhere. Nevertheless the continuous operating temperatures of most thermoplasts and thermoplastic matrix materials are still significantly lower than those of duroplastic matrix resins. At present only very high quality thermoplastics which are up to ten times more expensive than duroplastics allow continuous use at operating temperatures similar to these.

Table 7 provides an overview of the current manufacturing technologies for fiber-reinforced plastics.

Table 7: Manufacturing Technologies for Fiber-Reinforced Plastics (BMC = Bulk Molding Composite, SMC = Sheet Molding Composite, GMT = glass matting-reinforced thermoplastic)

Manufacturing Process	Material	Industrial Use	Production Speed
Injection molding	Thermoplastics, duroplastics, BMC	Very widespread	High
Hot pressing	Resin matting, SNC, prepreg	Widespread	Medium
Hot forming	Thermoplastic semifinished products, long fiber-reinforced, GMT	Beginning	Relatively high
Winding	Fiber-reinforced multilayer laminates, duroplastics	Beginning	Relatively low
Pultrusion	Unidirectional continuous laminates	Beginning	Medium

For the mass production of components, for example those for use in automobiles, cost-efficient processes are required with fast manufacturing and cycle rates and the highest possible level of automation. This affects the hardening times of the matrix resins, which must have both good fiber impregnation and good fiber bundle impregnation. One interesting advantage of CFK (carbon fiber-reinforced plastic) components is that their thermal expansion can be reduced to nothing by aligning the fibers in a particular direction. A disadvantage of these materials in high performance applications is their inadequate impact and fracture resistance; it is true that the component does not suddenly fail when subjected to impact, but damage occurs to the laminate structure

which reduces the strength of the material. The reason for this brittleness is the inadequate ductility of the composite. In recent years there has been a 2 percent improvement in the breaking elongation of high performance carbon fibers, but this ductility in the reinforcing fibers can nevertheless only be fully utilized if the matrix ductility is two to four times greater than that of the fibers and if adequate bonding ensures a satisfactory transfer of energy between fiber and matrix. To fully exploit the strength ratings of high performance carbon fibers, polymer matrix systems must be developed that bond well to the fiber and have adequate ductility.

For example, a project begun in 1986 studied the application of the strip lay-up technique as a mass production

process for non-dynamically balanced components, taking an engine mounting as a planar automobile component. Similarly, the winding method was applied to a dynamically balanced part, a prototype cardan shaft. Tests on the engine mounting prototypes and acoustic scanning showed that the strip lay-up technique should be feasible as a mass production process for a polymer composite material. A production feasibility study on the automated mass production of winding structures was carried out and data was collected for computer-aided component design. Parameter studies were done on high-speed winding of vinyl ester filament prepgs with filament speeds up to 2.5 meters per second. A seven-axle industrial robot was incorporated into a winding unit and equipped with the peripherals for prepg processing.

The purpose of the CARMAT 2000 development, begun in 1987, is to use polymer composites as widely as possible in automobiles. The overall goal is to gather basic data for large scale mass production on the basis of a prototype series which incorporates as many of the existing components as possible into an integral component, using new materials with good acoustic and electromagnetic scanning properties. Within this EUREKA [European Research Coordination Agency] project, in which companies from France, the Netherlands, Italy, and Spain are also involved, the FRG participants are seeking prototype solutions to problems in manufacturing bumpers, side doors, and floor units, among other things. The first components have been made and are being tested.

Polymer matrix materials better adapted to ductile carbon fibers are needed to develop composite materials with high temperature dimensional stability, resistance to environmental factors, and a considerably greater work capacity. A program begun in 1985 involves wideranging projects to synthesize new duroplasts; some of these projects have now been completed. The newly-developed resin systems show considerably better energy at break, flexural strength, and humidity absorption properties. The attempt to adapt the matrix resins to the expansion ratings of carbon fibers was partially successful.

The development and characterization of high performance polymer composites with thermoplastic matrices requires an extensive knowledge of basic data on the fiber/matrix interface and materials processing. In a project that began in 1988 a number of trial composites (various unidirectional and 0/90° carbon fiber-reinforced polymers: polysulfone, polyether sulfone, polycarbonate, polyether imide, and polyether ketone as matrix materials) were produced and tested. A comprehensive experimental study of the bonding properties of commercial carbon fibers in thermoplastics was initiated. Production of prepgs was improved on a laboratory scale and positive results were achieved in parameter studies of film molding techniques and in postformability tests on thermoplastic carbon fiber composites.

Work began in 1985 on developing nondestructive procedures for testing components made from composites to ensure quality control during production and detection of damage during maintenance. The goal was to apply holographic interferometry in testing large components. Delaminations near the surface of carbon fiber-reinforced plastic (CFK) sandwich components were detected down to a corner length of 10mm. Delaminations were reliably detected in CFK structures with integrated reinforcing elements at depths of up to 20mm. The areas around impact faults are marked by interference patterns which are still difficult to interpret.

Another project on testing composite components was started in 1987 and appears promising judging by the initial results. As part of a EUREKA project in which a French and a Spanish partner are also participating, research is being carried out on the potential of neutron radiography for detecting small faults deep within the component.

Composite Materials With Metallic Matrices

[Text] As with polymer composites, the main objective in developing metallic matrices is the technological exploitation of the increase in strength achieved with embedded reinforcing elements. Lightweight metals such as aluminum and magnesium have not been usable so far in high temperature applications because of their low thermal resistance. For example, the tensile strength of aluminum alloys drops at 200° C to about 50 percent of its value at room temperature. The main objective in developing composites with metallic matrices is therefore to raise their thermal resistance by embedding particles, whiskers, or fibers. The development of composite materials using aluminum and magnesium as the matrix and using ceramic fibers, whiskers, and particles as reinforcers was begun in 1986. Various techniques were tried: embedding chipped fibers at atmospheric pressure in molten aluminum by continuous casting, embedding chopped fibers, whiskers, and particles in molten magnesium at atmospheric pressure, and the production of material in reshappable blocks. The technical qualities of the materials were tested in prototype components for piston engines (connecting rods, cores for piston pins, and spring plates). So far the material meets requirements to only a limited extent. Improved processing techniques should remove the prime causes of the faults noted (for example, damage to fibers during continuous casting).

The goal of another project, which was launched in 1986, is to reinforce long and short fibers of AlSi and AlMg alloys for automobile components. This will also include a feasibility study for subsequent mass production. The promising laboratory results achieved with long fiber-reinforced parts were successfully reproduced in large scale production. However, the mechanical processing of the material is still proving problematic.

A project begun in 1985 is aimed at combining ceramic and metallic matrix materials: Al_2O_3 fiber-reinforced aluminum materials with a bonded ceramic surface layer for combustion engine pistons will be produced using a

graded structure. Initial results have been obtained. A purification technique was used to achieve considerable improvement in the quality of reinforcing fibers which to date had only been obtainable abroad. Components have successfully passed the first tests.

Composite Materials With Ceramic Matrices

[Text] In program area 1, we reported on ceramic materials, their technological and scientific attractiveness, and also on the problems involved in their use. One possible way of eliminating the disadvantages associated with ceramics is to embed particles, whiskers, or fibers in the ceramix matrix. These composites retain the positive properties typically associated with ceramics, at the same time offering better thermal shock resistance. In addition, their brittle fracture behavior improves. Embedded elements can have the same chemical composition as the matrix, as when silicon carbide fibers are embedded in a silicon carbide matrix (SiC/SiC).

A concentrated effort to develop highly tensile fiber-reinforced composites with ceramic matrices was made in the FRG at the beginning of the Materials Research Program by two strong teams recruited from industry and research institutes. Whiskers and short and long fibers will be used to reinforce ceramic materials (Al_2O_3 , SiC, Si_3N_4 , glass ceramics, and glass). Various techniques for coating the reinforcing elements were tried and the composites produced were subjected to microanalytical, microstructural, and fracture tests.

In spite of the pure nature of this line of research, remarkable results have already been achieved.

SiC fiber reinforcement fiber-reinforced cordierite glass ceramics yielded flexural strength levels of up to 750 MPa at fracture toughness levels of $17\text{-}20 \text{ MNm}^{-3/2}$. Duran glasses reinforced with SiC fibers gave flexural strength values of 1000 MPa at fracture toughness levels of $34 \text{ MNm}^{-3/2}$.

The use of SiC fiber-reinforced silicon carbide in diesel engines is being investigated in a EUREKA project started with France in 1985. Components were tested for their static and dynamic behavior and were tried out in engines. Results so far are highly positive and the components have come through the trials successfully. Further work must still be done on some useful findings, and the sealing or coating of material surfaces must still be improved.

A project was started in 1986 to improve the oxidation resistance of carbon fiber-reinforced carbon (CFC). Because of its high thermal stability (it can be used in a vacuum up to 2000°C without loss of strength) and its great thermal shock tolerance and toughness, this material is expected to play a key role in the construction of future high performance power units. However, its poor resistance over long periods of use in oxidizing environments presents a significant problem. Interim data from

research and development in this area show that a decisive reduction in susceptibility to oxidation can be achieved by impregnation with SiC. Thus, for example, after a 15-hour CVI treatment with SiC, CFC samples showed only a minimal tendency to oxidize at a temperature of up to approximately 1300°C .

Improved Reinforcing Fibers

[Text] Apart from the monocrystal whisker, which has only been known since about 1960, the strength of materials as calculated from the binding energy of their atoms can be put to only a limited practical use. A paradox postulated by Griffiths runs: "A material in fiber form is several times stronger than the same material in a different form; the thinner the filament, the greater the strength." This principle is based on the fact that the probability of fracture-inducing volume defects in a material diminishes as the volume is reduced and that there is little probability of fracture-inducing surface defects in fibers, which are usually spun from a molten mass or a solution.

Table 8 provides a breakdown of the strengths of various materials, comparing strength values achieved to date with those that are theoretically possible. Research and development on reinforcing fibers focuses on the development of techniques for the continuous production of the thinnest possible fibers with high tensile strength, using currently available or new materials. A second important step is the production of a fiber surface that forms the optimum interface bond with the matrix in which it is embedded for the use for which the material is intended. According to the various applications, the bond may have to be stable and light or weak and shifting. Apart from fibers, other examples of reinforcing materials are particles, thin films, or whiskers which are often irregularly embedded in the matrix, thus producing materials with a less marked anisotropic strength profile than is the case with fiber reinforcement.

Table 8: Strengths of Various Materials

Material	Strength (measured in GPa)		
	Solid Material	Fiber (elongated)	Theoretically Possible
AR	0.6	0.8	3.8
Cu	1.2	3.0	6.2
B	(0.3)	3.4	17
Fe	1.4	4.1	11.2
Polyethylene	0.03	1.0	25
Polyamide	0.08	0.85	25
Aramid		3.0	25
Glass	(0.1)	4.0	11
Al_2O_3	0.2	1.6	26
Carbon	(0.1)	3.0	35

Two examples of development projects are outlined below. The first, which was begun in mid-1986, is developing new techniques to produce high-performance carbon fibers using cheap raw materials. Carbon fibers

are currently produced mainly from polyacrylonitrile (PAN), which is a relatively expensive method due to the raw material and processing costs. Coal tar pitch, which is relatively cheap, could be an alternative, but so far this method has only produced lower quality carbon fibers. Laboratory studies aimed at using intermediate phase pitch to make carbon fibers with a tensile strength of over 2 GPa and a break elongation of at least 1 percent were begun and the requisite improvements in the process for preparing intermediate phase pitch as a raw material were achieved. A technical institute for the production of intermediate phase pitch was put into operation and filament yarns were spun using a laboratory spinning machine. Results achieved so far suggest that the carbon fibers of the quality outlined above can be attained.

The second research and development project, which started in 1986, is focused on characterizing the carbon fiber surface using suitable methods and on varying the surface of the fiber to acquire the mechanical characteristics ideally required of the composite material. The results of the studies concluded so far include reproducible data on the nature of certain molecule groups on the surfaces of conventional carbon fibers. Samples of composites were produced by the wet winding technique and the effect of various fiber types with different surface structures in transverse tensile strength was determined. Work on silicon carbide and silicon nitride-based ceramic fibers was begun early in 1988, so no measurable results are available yet. If the work continues on schedule, FRG manufacturers could also be meeting the demand for such ceramic fibers in a few years.

New Structural Concepts

[Text] Seven research organizations, four research institutes, and three companies are working on the new structural concepts area within this program.

Composite materials have anisotropic strength properties, especially when embedded reinforcing elements are aligned in a particular direction. This causes problems both when force is applied to components made of composites and in component assemblies. New structural concepts for the production of composites capable of withstanding multidirectional stress, technically and economically viable production processes, and new

methods of calculation will overcome these problems and broaden the range of applications for composites.

Three-dimensionally reinforced fiber composites using 3D fiber structures have been under study since 1986. The goal is to improve impact resistance and susceptibility to delamination, and to achieve considerable reduction in production costs by improving component manufacturing techniques. Recently-developed techniques for producing sandwich constructions with a variable structure have proven effective. Successful impregnation and hardening tests on matrix materials confirm their favorable impact resistance and low susceptibility to delamination. Comparisons of the impact resistance properties of 3D glass fiber composite material with 2D reinforcement have not yet yielded conclusive results.

Promising projects on new structural concepts have been started under the program. Since such concepts point the way to future composites, there are plans to increase work on this topic.

Overall costs and the BMFT share for composite materials projects started by June 1988: Overall costs: DM219.8 million BMFT share: DM116.4 million

Distribution of Resources Among R&D Topics

Improved reinforcing fibers	9%
Composites with polymer matrices	45%
Composites with metallic or ceramic matrices	41%
New composite structures	5%

Composite Materials

[Text]

Table 1: Total Costs and the BMFT Share in Projects Started by 30 June 1988

Subject Areas	Total Costs (in millions of DM)	BMFT Share
Ceramics	251.0	124.4
Powder metallurgy	93.7	57.6
Metallic high temperature materials	176.0	99.5
New polymers	199.6	103.3
Composite materials	219.8	116.4
Total	940.1	501.2

Table 2: Distribution of Funding Among Industry and Institutes

Subject Area	Industry	Institutes	Total
Ceramics	81.9	42.5	124.4
Powder metallurgy	36.8	20.8	57.6
Metallic high temperature and special materials	58.5	41.0	99.5
Polymers	79.9	23.4	103.3
Composite materials	96.0	20.4	116.4

Table 2: Distribution of Funding Among Industry and Institutes

Subject Area	Industry	Institutes	Total
Total	353.1	148.1	501.2
Funding included in subcontracts from industry to institutes (DM19.7 million)	-19.7	+19.7	
Total	333.4	167.8	501.2
Percent	66.5%	33.5%	100.0%

Table 3: BMFT Grant Distribution, Broken Down According to Company Size

Subject Area	Large Companies	Small to Medium-Sized Companies	Total
Ceramics	75.1	6.8	81.9
Powder metallurgy	26.1	10.7	36.8
Metallic high temperature and special materials	52.2	6.3	58.5
Polymers	76.7	3.2	79.9
Composite materials	84.7	11.3	96.0
Total	314.8	38.3	353.1*

*includes DM19.7 million in grants passed on to research institutes in subcontracts

Table 4: Distribution of Research Grants Among Research Institutes (in millions of DM)

Subject Area	Universities	Fraunhofer Institutes	Max Planck Institutes	Major Research Institutes	Others	Total
Ceramics	24.9	4.6	12.8	0.2		42.5
Powder metallurgy	12.6	0.8	2.3	3.7	1.4	20.8
Metallic high temperature and special materials	17.0	1.7	11.0	7.2	4.1	41.9
Polymers	10.8		11.2		1.3	23.4
Composite materials	11.7	2.6	0.6	3.3	2.2	20.4
Total	77.2	9.7	38.0	14.4	9.0	148.1
Grants included in subcontracts from industry to institutes						19.7
Total Grants to Institutes						168.0

Tables 5 and 6 give an overview of the current world markets for structural ceramics and for high stress composite materials. It can be seen that in 1985 worldwide sales of structural ceramics in the high temperature sector was very small: \$505 million. In the same year, world sales of composite materials for the vehicle industry, mechanical engineering, and medicine were approximately the same at \$545 million. In assessing the growth

potential for these materials in the application areas mentioned above, the Japanese predict dramatic rates of increase, while the United States and Western Europe give a considerably more conservative estimate. MITI [Japanese Ministry of International Trade and Industry] predicts sales of approximately \$10 billion for the Japanese market alone in the year 2000, including ceramics applications in electronics which are still dominant.

Table 5: Structural Ceramics Sales (in millions of \$, 1985)

Product	Worldwide	Japan	United States	Europe
Engine parts	30	18	10	2
Tool components	75	20	15	30
Wearing parts	300	100	100	100
Bioceramics	40	10	10	20
Others	60	40	10	10
Total	505	188	145	162

Source: R.N. Katz, Feldmuhle/Kyocera 1987

Table 6: Sales of High Performance Composite Material Components (in millions of \$, 1985)

Regional Markets	
United States	1,350
Western Europe	550
Japan	200
Total	2,100

Source: OTA [Office of Technology Assessment] study, "New Materials by Design," June 1988

Table 7: International Participation in the Materials Research Program

Regional Markets	Partner Countries
COST [European Cooperation in the Field of Scientific and Technical Research] 501: materials for generating energy; COST 502: Corrosion in building research; COST 503: powder metallurgy; COST 504: foundry technology; COST 505: materials for steam turbines. In preparation: COST 507: computer-aided phase diagrams of light metal alloys	Belgium, Denmark, France, Great Britain, Italy, Finland, Norway, Sweden, Switzerland, joint European Community research organizations
EUREKA Projects	
EU 13, CARMAT 2000	Netherlands, UK, France, Spain
EU 22, automatic integrated system for neutron radiography (DIANE)	France, Spain
EU 33, use of ceramics in gas turbines	France, Italy, Sweden
EU 42, light materials for transport systems	France
EU 47, development of ceramic materials for diesel engines	France
In preparation:	
Production of direct-cast thin films	France
Highly tensile steels for automobile construction	Sweden
Aluminum powder metallurgy	France
Ceramic gas turbines for automobiles	France, Sweden
Characterization and testing of ceramic powders and materials (IEA [International Energy Agency] agreement)	United States, Sweden
Polymer research, composite development, powder metallurgy	Israel
High temperature titanium alloys, titanium coatings, thermomechanical treatment of steels	PRC
Optimization of high performance welding techniques for steels	Finland
VAMAS—Versailles Project on Advanced Materials and Standards	Canada, France, Japan, United States, Italy, UK, Commission of the European Community

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